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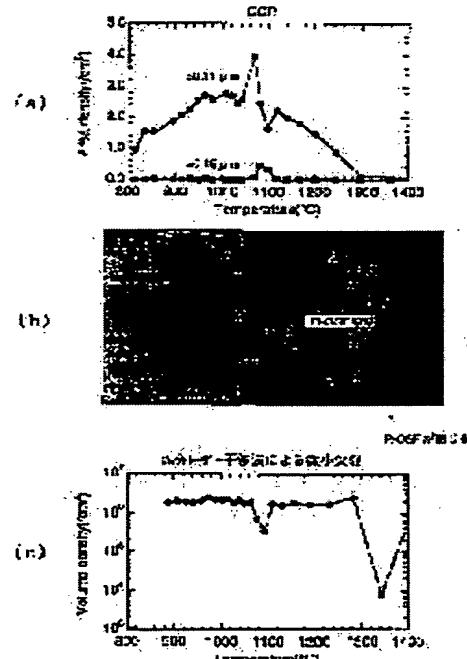
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IWASAKI TOSHIO**(54) SILICON SINGLE CRYSTAL AND ITS PRODUCTION****(57)Abstract:**

PROBLEM TO BE SOLVED: To provide a method for pulling up a crystal by which crystal originated particles(COP) and microdefects to be a cause of producing the COP are reduced in the crystal produced by the Czochralski method.

SOLUTION: This method for producing a silicon single crystal comprises annealing a crystal in a temperature region of the melting point to $\geq 1,300^{\circ}\text{C}$ for ≥ 400 min and pulling up the crystal (method 1), regulating the time for passing a temperature region of $\geq 1,350^{\circ}\text{C}$ to <60 min in addition to the method 1 (method 2), carrying out the pulling up of the crystal under conditions so as to provide >0.13 ratio (v/G) of the pulling up speed (v: mm/min) to the crystal

temperature gradient in the solidification interface (G: $^{\circ}\text{C}/\text{mm}$) in addition to the method 1 (method 3), annealing the crystal at $<1.0^{\circ}\text{C}/\text{min}$ crystal cooling rate in a temperature region of 1,100 to 1,000 $^{\circ}\text{C}$ in addition to the method 1 or 2 (method 4) and quenching the crystal at $\geq 1.0^{\circ}\text{C}/\text{min}$ crystal cooling rate in the temperature region of 1,100 to 1,000 $^{\circ}\text{C}$ in addition to the method 1, 2 or 3 (method 5).



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CLAIMS

[Claim(s)]

[Claim 1] The manufacture approach of the silicon single crystal characterized by carrying out crystal raising growth whose time amount which passes through the inside of a 1300-degree C crystal temperature field from the melting point is 400 minutes or more in manufacture of the silicon single crystal by the Czochralski method.

[Claim 2] The manufacture approach of the silicon single crystal characterized by carrying out crystal raising growth whose pass time in the crystal temperature field whose pass time of a 1350-degree C crystal temperature field is less than 60 minutes and 1350 degrees C - 1300 degrees C is 400 minutes or more from the melting point in manufacture of the silicon single crystal by the Czochralski method.

[Claim 3] The manufacture approach of the silicon single crystal characterized by pulling up in manufacture of the silicon single crystal by the Czochralski method on the conditions from which the crystal temperature gradient G (degree C/mm) in the pull-up rate v (a part for mm/) and a coagulation interface is set to $v/G > 0.13$, and carrying out crystal raising growth whose pass time in a crystal temperature field 1300 degrees C or more is 400 minutes or more.

[Claim 4] The claim (1) to which the cooling rate of a 1100 to 1000 degrees C temperature requirement is characterized by 1.0-degree-C being the following by /, and the crystal manufacture approach given in (2).

[Claim 5] The claim (1) to which the cooling rate of a 1100 to 1000 degrees C temperature requirement is characterized by 1.0-degree-C being above by /, and the crystal manufacture approach given in (2).

[Claim 6] The silicon single crystal with which it is the silicon single crystal manufactured by the approach of of a claim (1) (2) (3) thru/or (5), and big COP 0.13 micrometers or more does not exist.

[Claim 7] The silicon single crystal whose surface density of COP 0.11 micrometers or more is two or less [0.1 //cm] in the wafer by which mirror plane processing was cut down and carried out from the silicon single join manufactured by the approach of of a claim (1) (2) (3) (4) thru/or (5).

[Claim 8] The silicon single crystal whose volume density of the minute crystal defect which forms COP in the silicon single join manufactured by the approach of of a claim (1) (2) (3) (4) thru/or (5) is three or less [5x10⁵ //cm].

[Claim 9] The silicon single crystal whose minute crystal defect volume which is the silicon single crystal manufactured by the approach of of a claim (1) (2) (3) (4) thru/or (5), and forms COP is three or less [1012nm] to the unit volume (1cm³) of a host phase.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]**[0001]**

[Field of the Invention] This invention relates to the silicon single crystal manufactured by the Czochralski method (the following, CZ process) which reduced the crystal defect sharply, and its manufacturing method.

[0002]

[Description of the Prior Art] Since CZ silicon single crystal has the description which was [be / crystal reinforcement / high] excellent, it is widely used as an ingredient for LSI from before. Manufacturing-technology development of CZ silicon single crystal which is excellent in device properties, such as improvement in dependability of gate oxide and PN-junction leak reduction, and the defect in separation between components cannot produce easily was desired with increase of an MOS device degree of integration in recent years. Especially COP is the minute pit which exists in the silicon wafer front face after mirror polishing, it was shown clearly recently that the minute defect used as the cause of generating this pit itself and pit caused [of the device property described previously or separation between components] a defect, and it was anxious for that reduction.

[0003] After washing a wafer front face after polish in a silicon wafer processing process with the mixed liquor (hydrogen peroxide: ammonia : water = 1:1:5) of aqueous ammonia and hydrogen peroxide solution (called SC-1 washing), the pit resulting from a minute defect forms in a wafer front face, and if it measures at a laser particle counter, it will be detected [COP / (Crystal Originated Particle)] as particle with intrinsic particle. It is referred to as COP in order to distinguish such a pit from intrinsic particle. About this reduction approach, that reduction technique was conventionally known with regards to improvement in the oxide-film pressure-proofing in CZ silicon single crystal manufacture. The approach of holding to a 1200 degrees C - 1420 degrees C temperature field for 1 hour or more, and carrying out crystal pulling to it in JP,6-279188,A, is indicated. In JP,8-2993,A It is made for the time amount which passes the pyrosphere to 1200 degrees C to become 200 minutes or more. In the approach and JP,8-157293,A which the time amount which passes through a 1200 degrees C - 1000 degrees C low-temperature region makes 150 or less minutes The approach of making time amount which makes time amount which passes the pyrosphere to 1200 degrees C less than 200 minutes, and passes through a 1200 degrees C - 1000 degrees C low-temperature region 130 or less minutes is indicated.

[0004] In these conventional techniques this invention persons The various crystals which changed the cooling conditions of the crystal under pull-up extremely are raised. Again With the experiment which holds a crystal in a raising furnace, only only not only in COP observation of the wafer immediately after polish / washing How to repeat washing and observation, to remove dead genuineness particle, and to evaluate the volume density of only the defect of a COP generating cause, And it is based on the observation experiment which evaluates the total amount of the point defect which constitutes the minute defect which measures simultaneously not only the volume density of the minute defect of a COP generating cause but size with an infrared laser interference method, and causes COP generating. There is no effectiveness in reduction of the minute defect COP and leading to COP generating at maintenance of a 1200 degrees C - 1300 degrees C temperature field. Furthermore, the crystal pass time in the temperature field also found out that remarkable reduction of the minute defect of COP and a COP generating cause was not seen in about 60 - 200

minutes.

[0005] Therefore, the crystal pulling approach of reducing the minute defect which becomes COP and a COP generating cause notably in the crystal under pull-up, furthermore, R-OSF (a Ringlikely distributed Oxidation-induced-Stacking-Faults:ring-like distribution stacking fault --) which occupies most which is circulating in the present commercial scene Bibliography : It is related with the crystal with which magazine "application physics" 57 volume, 1541 pages, the crystal that a field did not exist in the field in 1988, but has disappeared in the edge section, or the R-OSF field has not disappeared centering on a wafer. The optimal approach of reducing extremely the minute defect COP and leading to COP generating did not exist. Moreover, in the crystal which a R-OSF field does not exist in a wafer side, and has not disappeared centering on a wafer, the crystal which reduced extremely the minute defect COP and leading to COP generating did not exist, either.

[0006]

[Problem(s) to be Solved by the Invention] In the crystal with which this invention is manufactured by the Czochralski method (the following, CZ process) The crystal pulling approach of reducing the minute defect which becomes COP and a COP generating cause notably, Furthermore, it is related with the crystal with which the crystal or R-OSF field which occupies most which is circulating in the present commercial scene, and where a R-OSF field did not exist in the field, but has disappeared in the edge section has not disappeared centering on a wafer. It is related with the silicon single crystal which reduced extremely the minute defect COP and leading to COP generating, and its manufacturing method.

[0007]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, in this invention, about the crystal under pull-up, crystal temperature is a field 1300 degrees C or more, and crystal pulling growth is performed on the conditions for 400 minutes or more held (approach 1). Furthermore, in order to remove a R-OSF field to a crystal edge outside and to reduce the minute defect of COP and a COP generating cause, pass time of a 1350-degree C crystal temperature field is made into less than 60 minutes from the melting point, and crystal pulling which holds a 1350 degrees C - 1300 degrees C crystal temperature field 400 minutes or more is performed (approach 2). Or in order to remove a R-OSF field to a crystal edge outside and to reduce the minute defect of COP and a COP generating cause, when setting a pull-up rate to v (a part for mm/) and considering as the crystal temperature gradient G in a coagulation interface (degree C/mm), crystal raising growth is carried out so that the time amount which pulls up on the conditions used as $v/G > 0.13$, and passes through the area within crystal temperature 1300 degrees C or more may become 400 minutes or more (approach 3). furthermore -- or (approach 1) (approach 2) -- or (approach 3) -- in addition, in order to make still smaller minute defect density of COP and a COP generating cause, the cooling rate of a 1100 to 1000 degrees C temperature requirement is made into the following by 1.0-degree-C/, and is annealed (approach 4). moreover -- or (approach 1) (approach 2) -- or (approach 3) -- in addition, in order to make still smaller COP and minute defective size of a COP generating cause, the cooling rate of a 1100 to 1000 degrees C temperature requirement is considered as the above by 1.0-degree-C/, and is quenched (approach 5).

[0008] The silicon single crystal with which big COP 0.13 micrometers or more does not exist is obtained by manufacturing by the approach of this invention approach 1 2 3 thru/or 5. Moreover, in the wafer front face which cut and processed [mirror plane] it from the silicon single crystal, the silicon single crystal whose COP 0.11 micrometers or more is two or less [0.1 //cm] can be manufactured by the approach of this invention approach 1 2 3 4 thru/or 5. According to the manufacture approach of 1-5 of this invention, the volume density of the minute crystal defect which forms COP which exists in a silicon single crystal is three or less $5 \times 10^5 / \text{cm}^3$. Moreover, the silicon single crystal whose point defect concentration which constitutes the minute defect concerned is three or less [1012nm] to the unit volume (1cm³) of a host phase and which usually reduced single or more figures minute defect density and point defect concentration compared with the crystal conventionally is obtained. Device properties, such as the withstand voltage property of an insulating oxide film and a P/N leak property, are also excellent.

[0009]

[Embodiment of the Invention] this invention persons raise the various crystals which changed the

cooling conditions of the crystal under pull-up extremely. With the experiment which holds a crystal in a raising furnace, only moreover, only not only in COP observation of the wafer immediately after polish / washing How to repeat washing and observation, to remove dead genuineness particle, and to evaluate the volume density of only the defect of a COP generating cause, And it is based on the observation experiment which evaluates the total amount of the point defect which constitutes the minute defect which measures simultaneously not only the volume density of the minute defect of a COP generating cause but size with an infrared laser interference method, and causes COP generating. It discovered that the following relation between formation of the minute defect COP and leading to COP generating and crystal cooling conditions was. That is, when the generating behavior of the minute defect which pulls up the crystal under pull-up, is the middle, pulls up, slows down a rate to a idle state extremely mostly, holds a crystal in a raising furnace, and becomes COP and a COP generating cause was investigated, a result as shown in drawing 1 was brought. The volume density of the minute defect leading to [which was measured with the infrared laser interference method] COP generating is shown in an X-ray topogrph photograph to show the surface density of COP 0.11 micrometers or more in drawing 1 (a), and show a R-OSF field in drawing 1 (b), and drawing 1 (c) as a function of the retention temperature of the crystal under pull-up, respectively. The minute defect COP and leading to COP generating of a reduction ***** field is 1300 degrees C or more in a zero level and volume density at surface density at three or less 104-/cm so that clearly from drawing 1 (a) and (c). Moreover, in the X-ray topogrph photograph of drawing 1 (b), it is clear from the melting point that the part's which had the 1350-degree C temperature field held the R-OSF field has closed and disappeared in the crystal center section. Therefore, in order to reduce the minute defect used as the cause of generating COP or COP, 1300 degrees C or more need to be cooled slowly, but if 1350 degrees C or more are annealed too much, a R-OSF field comes to exist in a crystal center side. Moreover, it is admitted that volume density decreases in a 1100 degrees C - 1000 degrees C temperature requirement in drawing 1 (c), and a temporary reduction with a COP surface density of 0.11 micrometers or more and a temporary increment are accepted in this temperature requirement in drawing 1 (a). Since the location where the COP surface density of 0.11 micrometers or more decreases temporarily is in agreement with the location which big COP 0.16 micrometers or more increases, in this temperature field, it turns out that the minute defect leading to COP generating grows. Especially a big minute defect grows by the elevated-temperature side more, and a small minute defect grows by the low temperature side. Consequently, it is thought that a temporary increment in COP 0.11 micrometers or more is the field where the small COP generating cause minute defect grew. It is possible to control growth of the minute defect which causes COP generating from this result by raising the cooling velocity of this temperature field, and it is possible to reduce minute defect density further by annealing this temperature field.

[0010] Although the above was as a result of [by maintenance of the pull-up crystal in a raising furnace] minute defective observation, when it investigated the annealing effectiveness of the pyrosphere at the time of carrying out crystal growth with constant speed, without having actually pulled up in the raising furnace, and slowing down and holding an inner crystal, it obtained the result shown in drawing 2 . That is, drawing 2 (a) shows change of the number of COP 0.11 micrometers or more to the residence time of the crystal which passes through a temperature field 1300 degrees C or more, and drawing 2 R> 2 (b) shows the result corresponding to total amount change of the whole minute defective product leading to [over the residence time of the crystal which passes through a temperature field 1300 degrees C or more] COP generating, i.e., the point defect which constitutes the minute defect concerned. In this crystal, a R-OSF field is located in the crystal edge section, and does not exist in a central field. COP is a zero level mostly and by passing through a temperature field 1300 degrees C or more 400 minutes or more shows that the total amount of the point defect which is the minute defective component made to generate COP decreases a figure single [about] so that clearly from drawing 2 R> 2.

[0011] Although elevated-temperature annealing is required in order to control formation of the minute defect COP and leading to COP generating from the above result, the annealing time amount which an effective temperature requirement is 1300 degrees C or more, and is needed is 400 minutes or more.

[0012] Furthermore, it turned out that the manufacture conditions of a crystal that the crystal or R-

OSF field which occupies most which is circulating in the present commercial scene and where a R-OSF field did not exist in the field, but has disappeared in the edge section has not disappeared centering on a wafer are the following two conditions. That is, if annealing temperature is made into 1350 degrees C or more, in connection with annealing time amount, a R-OSF field will come to enter into a core side from the crystal edge section. In this invention, annealing time amount 1350 degrees C or more became clear [that less than 60 minutes is desirable] as conditions which do not vanish a R-OSF field in the crystal center section. as another conditions on which a R-OSF field does not disappear in the crystal center section -- the ratio of the crystal temperature gradient (G:degree C/mm) of a pull-up rate (v:mm/) and a coagulation interface -- it turned out that what is necessary is just to perform crystal pulling on the conditions to which v/G becomes larger than 0.13. the ratio of the crystal temperature gradient (G:degree C/mm) of this pull-up rate (v:mm/) and a coagulation interface -- although it was indicated by v/G at JP,7-257991,A that the generating location of a R-OSF field changes, in this invention, it turned out that the crystal pulling conditions on which a R-OSF field does not disappear in the crystal center section are conditions to which v/G becomes larger than 0.13.

[0013] About the crystal cooling conditions by the side of the low temperature for COP defective control, with regards to growth of the minute defect from which the effectiveness causes COP generating in JP,8-2993,A, growth of the minute defect concerned is controlled for the one where cooling velocity is larger, and although growth progresses by making cooling velocity small, it is indicated that a consistency can be reduced. However, in this invention, when performing enough elevated-temperature annealing of 1300 degrees C or more, it was found out for the first time that the minute defect from which the quenching rate in a field becomes COP and a COP generating cause even a minute in about 1.0 degrees C /whenever [1100 degrees C - 1000 degrees C low-temperature] can fully be reduced. Furthermore, when fully performing elevated-temperature annealing of 1300 degrees C or more, in the field, the minute defect which becomes a big COP generating cause by [1.0 degree-C] cooling slowly with the following cooling velocity by /did not grow, but bringing about much more consistency fall rather was also found out for the first time whenever [1100 degrees C - 1000 degrees C low-temperature]. Moreover, it cannot be overemphasized by combining quenching (a part for 1.0-degree-C/[A cooling rate] above) of a field whenever [this invention sufficient elevated-temperature annealing / of 1300 degrees C or more /-, and 1100 degrees C - 1000 degrees C low-temperature] that the depressor effect of COP generating by reduction of COP size becomes remarkable. The effectiveness of a 1100 degrees C - 1000 degrees C temperature field Are effective in reducing the minute defect density of COP and a COP generating cause by promoting condensation of the point defect which is the component of the minute defect which becomes COP and a COP generating cause when a cooling rate is annealing of the following by 1.0-degree-C/. When a cooling rate is the above quenching by 1.0-degree-C/, it is effective in reducing the size of the minute defect of COP and a COP generating cause by controlling condensation of the point defect which is the component of the minute defect which becomes COP and a COP generating cause conversely.

[0014] By this invention approach 1, formation of the minute defect concerned is reduced by diffusing the atomic hole which is the main point defect which constitutes the minute defect COP and leading to COP generating from annealing the crystal which installs a temperature control function in a crystal raising furnace, and can be pulled up in a temperature field 1300 degrees C or more 400 minutes or more to the method of the outside of a crystal, and decreasing it.

[0015] By this invention approach 2, in addition to an operation of an approach 1, in order to make it a R-OSF field not enter into a crystal center side, time amount which passes through a temperature field 1350 degrees C or more is made into less than 60 minutes.

[0016] this invention approach 3 -- an operation of an approach 1 -- in order [in addition,] to make it a R-OSF field not enter into a crystal center side -- pulling up -- the ratio of the crystal temperature gradient (G:degree C/mm) of a rate (v:mm/) and a coagulation interface -- v/G performs crystal pulling on the conditions which become larger than 0.13.

[0017] this invention approach 4 -- an operation of an approach 1, an approach 2, or an approach 3 -- in addition, in order to reduce the minute defect density which becomes COP and a COP generating cause further, a 1100 to 1000 degrees C temperature field is annealed by the following by crystal

cooling rate/of 1.0 degrees C.

[0018] this invention approach 5 -- an operation of an approach 1, an approach 2, or an approach 3 -- in addition, in order to reduce the minute defective size which becomes COP and a COP generating cause further, a 1100 to 1000 degrees C temperature field is quenched above by crystal cooling rate/of 1.0 degrees C.

[0019]

[Example] Although the example of this invention is given and explained below, it cannot be overemphasized that this invention is not what is restricted by the publication of these examples.

[0020] In advance of an example, the evaluation approach of the existence of a R-OSF field and the device property evaluation approach are explained to the minute defect and pan used as COP of a silicon single crystal and the COP generating cause which were manufactured by this invention below.

[0021] After COP carried out wafer processing of the manufactured crystal and washed it by SC-1 penetrant remover of ammonia:hydrogen-peroxide:water =1:1:5 after mirror polishing, it measured the number of particle at the laser particle counter LS 6000. Furthermore, in order to remove intrinsic particle and to evaluate the volume density of only the defect of a COP generating cause, SC-1 washing and particle measurement were repeated 10 times, and it asked for volume density from the increment. The minute defect leading to COP generating measured a consistency and size with the measuring device (OPP:Oxygen Precipitate Profiler) by the infrared laser interference method. In order to generate OSF, about the existence of formation of a R-OSF field, it observed with the X-ray topograph after 90-minute heat treatment in the oxidizing atmosphere at 1100 degrees C.

[0022] Oxide-film pressure-proofing was performed by investigating the electrical property of the gate oxide (insulating oxide film) which is 25.0nm silicon dioxide film which formed the MOS diode on the silicon wafer sample which performed mirror plane processing, and was formed in the 1000-degree C desiccation oxygen ambient atmosphere. The average electric field impressed to gate oxide in case the current density which flows through an oxide film is 1microA/cm² were measured. Especially when the average electric field are 8.0 or more MV/cm, it is the field where the crystal defect which is called an intrinsic dielectric-breakdown field (C mode field), and causes proof-pressure degradation does not exist. Therefore, in proof-pressure characterization, a silicon wafer with many rates to the total of the number of the MOS diode (it is in a C mode field) which average electric field destroy by cm in 8.0MV /or more is the crystal which was excellent in the proof-pressure property.

[0023] Although an example and the example of a comparison are described below, the specifications of the crystal pulled up and raised are a conduction type:p mold (boron dope), the object (diameter of 160mm) for diameter of crystal:6 inches, resistivity:10 ohm-cm, oxygen density:8.5 - 9.5x10¹⁷ atoms/cm³ (it computes using the oxygen density conversion factor by Japan Electronic Industry Development Association), and carbon concentration:<1.0x10¹⁶ atoms/cm³ (it computes using the carbon concentration conversion factor by Japan Electronic Industry Development Association).

[0024] the volume density of the minute defect which causes [which was measured with COP measured at the laser particle counter LS 6000 of the wafer in an example 5 and the example 1 of a comparison, and the example 2 of a comparison, and an infrared laser interference method (OPP)] COP generating from the example 1 of this invention in Table 1, and oxide-film pressure-proofing -- the conclusion of the measurement result of each R-OSF's existence location is shown further.

[0025] The silicon single crystal manufacturing installation used for example 1 this invention is not limited especially if usually used for the silicon single crystal manufacture by the Czochralski method, and by this example, the manufacturing installation as shown in drawing 3 was used for it. The description of this Czochralski method silicon single crystal manufacturing installation installed the temperature controller, in order to form annealing ***** which crystal cooling temperature and a rate pattern go through a temperature region 1300 degrees C or more 400 minutes or more in raising conditions like [in drawing 6 (example 1)], and a crystal raising furnace, and a crystal passes. Heat insulation heat insulating material, heating heaters, etc., such as a graphite installed so that the silicon single crystal ingot by which raising training is carried out might be surrounded as a temperature controller, are effective.

[0026] This equipment was used and the silicon single crystal was raised on condition that the following. The volume density [of the minute defect leading to / which was measured with COP and the infrared laser interference method (OPP) of a wafer which were started from this single crystal ingot / COP generating], oxide-film pressure-proofing, and R-OSF's existence location was shown in Table 1. In these silicon wafers, COP 0.13 micrometers or more is a zero level, and even if it includes small COP 0.11 micrometers or more, it is reduced about single figure compared with the conventional technique by about ten pieces (0.05 piece/cm²). Moreover, the volume density of the minute defect COP and leading to COP generating is too reduced a single figure in three or less 1.0x10⁵/cm. A proof-pressure property is also very good as a result. In this crystal, the R-OSF field is contained from the edge inside a little for the effectiveness of annealing of 1350 degrees C or more.

[0027] The silicon single crystal was raised on condition that the following using the equipment of example 2 example 1. Although the pass time of crystal cooling temperature and raising conditions [like / in drawing 6 (example 2)] whose rate pattern is, i.e., a temperature field 1350 degrees C or more, was less than 60 minutes, it raised the silicon single crystal on conditions which go through a 1350 degrees C - 1300 degrees C crystal temperature field about 400 minutes, and are cooled slowly. The volume density [of the minute defect leading to / which was measured with COP and the infrared laser interference method (OPP) of a wafer which were started from this single crystal ingot / COP generating], oxide-film pressure-proofing, and R-OSF's existence location was shown in Table 1. Even if COP 0.13 micrometers or more is a zero level mostly like an example 1 and it includes small COP 0.11 micrometers or more also in these silicon wafers, compared with the conventional technique, it decreases about single figure below by 20 pieces (0.1 piece/cm²).

Moreover, the volume density of the minute defect COP and leading to COP generating is notably reduced on 1.0x10⁵/cm³ level. A proof-pressure property is also very good as a result. In this crystal, the R-OSF field has disappeared to the method of outside [edge / a crystal ingot edge or] writing the holding time 1350 degrees C or more as less than 60 minutes.

[0028] The silicon single crystal was raised on condition that the following using the equipment of example 3 example 1. the ratio of the temperature gradient G (degree C/mm) by the side of the crystal in the pull-up rate v (a part for mm/), and a coagulation interface -- it was made for v/G to become larger than 0.13 That is, in this example, the crystal temperature gradient in a coagulation interface was about 1.8 degrees C/mm, set the pull-up rate v as a part for 0.6mm/, and carried out crystal pulling (v/G in this case = 0.33). Furthermore, the silicon single crystal was raised on conditions which go through the crystal temperature field of 1300-degree-C or more ** about 400 minutes, and are cooled slowly. The crystal cooling temperature and the rate pattern in this case are among drawing 6 (example 3), it pulls up compared with an example 1, and since the rate is higher, the annealing time amount in an elevated temperature is short. however, heat insulation of near a coagulation interface and warming -- by changing conditions, a temperature gradient can be made still smaller than this example, or it is possible to extend an elevated-temperature field and annealing time amount can be lengthened. It is shown in the measurement result of the number of COP of the silicon wafer cut down from the single crystal ingot pulled up and raised in this example, the COP volume density for which it asked by repeat washing, the minute defect density leading to [which was searched for with the infrared laser interference method (OPP)] COP generating, and oxide-film pressure-proofing, and Table 1. Even if COP 0.13 micrometers or more is a zero level mostly like examples 1 and 2 and it includes small COP 0.11 micrometers or more also in these silicon wafers, compared with the conventional technique, it decreases about single figure below by 20 pieces (0.1 piece/cm²). Moreover, the volume density of the minute defect COP and leading to COP generating is notably reduced on 1.0x10⁵/cm³ level. A proof-pressure property is also very good as a result. In this crystal, the R-OSF field has disappeared to the method of outside [edge / crystal ingot].

[0029] In example 4 this example, the manufacturing installation as shown in drawing 4 was used. namely, the manufacturing installation shown by the example 1 and drawing 3 -- in addition, the upper part of the temperature controller shown in drawing 3 in order to also anneal a 1100 more to 1000 degrees C low-temperature temperature field with the following cooling rates by 1.0-degree-C-- further -- an incubation heat insulator and warming -- the incubation which consists of heaters, or warming -- equipment was installed. in addition, drawing 4 -- like -- new -- incubation or

warming -- even if it does not install equipment -- the temperature controller of drawing 3 -- a low, the upper part, i.e., crystal temperature, side -- you may extend . After holding a crystal temperature field 1300 degrees C or more 400 minutes or more using such a manufacturing installation, the 1100 more to 1000 degrees C temperature field was annealed with the following cooling rates by 1.0-degree-C/, and crystal pulling was performed. It is shown in the measurement result of the number of COP of the wafer cut down from this silicon single crystal ingot, the COP volume density for which it asked by repeat washing, the minute defect density leading to [which was searched for with the infrared laser interference method (OPP)] COP generating, and oxide-film pressure-proofing, and Table 1. In these silicon wafers, small COP 0.11 micrometers or more also has [COP 0.13 micrometers or more] the remarkable reduction effectiveness at below ten pieces (0.05 piece/cm²). However, it is the description that the increment in some [of big COP 0.13 micrometers or more] is seen according to the 1100 to 1000 degrees C annealing effectiveness. Moreover, the volume density of the minute defect COP and leading to COP generating is also the lowest on 5.0 to 8.0x10⁴/cm³ level. Consequently, a proof-pressure property is also very good. In this crystal, the R-OSF field has disappeared to the method of the outside of a crystal ingot edge or an edge.

[0030] In example 5 this example, the manufacturing installation as shown in drawing 5 was used. namely, the manufacturing installation shown by the example 1 and drawing 3 -- in addition, in order to raise a cooling rate, the cylindrical cooling system was installed above the temperature controller shown in drawing 3 in order to cool a 1100 more to 1000 degrees C low-temperature temperature field with the above cooling rate by 1.0-degree-C/. As a cooling system, a thermally conductive good graphite plate with large emissivity and a thermally conductive metal plate are effective, and are cooled by the cooling effectiveness by the convective heat transfer by the controlled atmosphere (this example argon gas) which carries out cooling of the radiant heat from a crystal, pulls up with the cooling system concerned further, and flows between intermediate crystals with the cooling system concerned. Forced cooling of the graphite plate and metal plate which are used as the cooling system concerned may be carried out using gas or a liquid. After holding a crystal temperature field 1300 degrees C or more 400 minutes or more using such a manufacturing installation, the 1100 more to 1000 degrees C temperature field was cooled with the above cooling rate by 1.0-degree-C/, and crystal pulling was performed. It is shown in the measurement result of the number of COP of the wafer cut down from this silicon single crystal ingot, the COP volume density for which it asked by repeat washing, the minute defect density leading to [which was searched for with the infrared laser interference method (OPP)] COP generating, and oxide-film pressure-proofing, and Table 1. In these silicon wafers, COP 0.13 micrometers or more is zero, and that of the reduction effectiveness is [small COP 0.11 micrometers or more] remarkable at below ten pieces (0.05 piece/cm²). Moreover, the consistency is reduced on 1.0x10⁵/cm³ level notably [the volume density of the minute defect COP and leading to COP generating]. Consequently, a proof-pressure property is also very good. In this crystal, the R-OSF field has disappeared to the method of the outside of a crystal ingot edge or an edge.

[0031] The example of the one example comparison of a comparison is an example of the conventional technique. The manufacturing installation used in the example 1 shown in drawing 3 and the same crystal growth equipment were used. However, a temperature controller is installed so that crystal cooling temperature and raising conditions [like / in drawing 6 (example 1 of a comparison)] whose rate pattern is, i.e., the elevated-temperature cooling conditions as [whose time amount from which the time amount held to a temperature field 1300 degrees C or more in a crystal raising furnace is held in about 40 minutes at 1200 degrees C or more is also about 80 minutes], may be realized, and it is silicon single crystal pull-up equipment [as / whose 1200 more degrees C - 1000 degrees C temperature field pass time is about 80 minutes]. Heat insulation heat insulating material, heating heaters, etc., such as a graphite installed so that the silicon single crystal ingot by which raising training is carried out might be surrounded as a temperature controller, are effective. However, as compared with an example 1, it is the description that the annealing crystal die length or time amount of an elevated-temperature field is short. The annealing time amount of an elevated-temperature field is about 100 or less minutes, and since the crystal cooling pattern of this example of a comparison is comparatively short, the temperature controller which controls cooling conditions does not necessarily need to be used for it, and it can realize it also by making the rate of crystal

growth, i.e., a pull-up rate, into a low speed.

[0032] The volume density [of the minute defect leading to / which was measured with COP and the infrared laser interference method (OPP) of a wafer which used this pull-up equipment and cut it down from the silicon single crystal ingot which carried out crystal growth / COP generating], oxide-film pressure-proofing, and R-OSF's existence location was shown in Table 1. In these silicon wafers, the number of it is 200 (1.0 piece/cm²), and when COP 0.13 micrometers or more includes small COP 0.11 micrometers or more, it is 500-piece (2.5 piece/cm²) extent. Moreover, there is volume density of the minute defect COP and leading to COP generating about three 1.0x10⁶/cm. The rate of surface ratio which destroys a proof-pressure property by the impression electric field of 8.0 or more MV/cm which are the indexes of the property is about 20%. the consistency of the minute defect from which the result of the example of these book comparison is the number of COP and a consistency, or the COP generating cause compared with the example of this invention -- all are very bad. Moreover, oxide-film pressure-proofing is not good, either.

[0033] In the example of the two example comparison of a comparison, crystal training was carried out using the equipment used in the example 1 of a comparison, and the same equipment. However, it is the description that the time amount from which the time amount which passes raising conditions [like / in drawing 6 (example 2 of a comparison)] whose crystal cooling temperature and rate pattern of this example of a comparison are, i.e., a temperature field 1300 degrees C or more, is held to a temperature field 1200 degrees C or more in about 120 minutes is about 220 minutes, and 1200 more degrees C - 1000 degrees C temperature field pass time is about 100 minutes.

[0034] The volume density [of the minute defect leading to / which was measured with COP and the infrared laser interference method (OPP) of a wafer which used this pull-up equipment and cut it down from the silicon single crystal ingot which carried out crystal growth / COP generating], oxide-film pressure-proofing, and R-OSF's existence location was shown in Table 1. In these silicon wafers, the number of COP 0.13 micrometers or more is 50 (0.25 piece/cm²), and the number of COP of 0.11 micrometers or more is 100-piece (0.5 piece/cm²) extent, and the number of COP is reducing it as compared with the example 1 of a comparison. Moreover, as for the volume density of the minute defect COP and leading to COP generating, compared with the example 1 of a comparison, reduction is too accepted a little in about three three to 1.0x10⁶/cm of 8.0x10⁵/cm. However, the number of COP which was realized by this invention also decreases the level almost near zero, or the volume density of the minute defect of a COP generating cause a single figure, and a proof-pressure property is also far inferior from the quality level whose rate of destructive surface ratio of 8.0 or more MV/cm is 60% or more.

[0035]

[Table 1]

| 冷却温度 パラメータ No. | >0.11 μm のCOP面数 6"シリコン | | >0.13 μm のCOP面数 6"シリコン | | 繰返洗浄によるCOP面数 >0.11 μm : /cm ² | 赤外線干熱法(OPP)による 欠陥密度 (>100nm : /cm ²) | 8.0MV/cm以上耐圧合格率 (%) | R-OSF存在位置 R-OSF外 |
|----------------------------------|---------------------------|-----|---------------------------|--------|---|---|------------------------|---------------------|
| | (1) | 12 | 0 | 9.0E4 | 8.0E4 | 70 | 15mm | |
| 実施例 1 (1) | 11 | 0 | 8.0E4 | 8.0E4 | 68 | 20mm | | |
| | 10 | 0 | 1.0E5 | 9.0E4 | 62 | 15mm | | |
| 実施例 2 (2) | 16 | 2 | 1.2E5 | 1.1E5 | 78 | 15mm | | |
| | 13 | 0 | 1.0E5 | 9.0E4 | 75 | 15mm | | |
| | 15 | 1 | 9.0E4 | 9.0E4 | 72 | 15mm | | |
| 実施例 3 R-OSFが結晶内部に 存在しない上条件 | 19 | 1 | 1.2E5 | 1.0E5 | 65 | 15mm | | |
| | 16 | 0 | 1.0E5 | 9.0E4 | 63 | 15mm | | |
| | 18 | 1 | 1.0E5 | 8.0E4 | 70 | 15mm | | |
| 実施例 4 (4) | 6 | 3 | 8.0E4 | 8.0E4 | 83 | 15mm | | |
| | 7 | 4 | 5.0E4 | 5.0E4 | 85 | 15mm | | |
| | 5 | 3 | 6.0E4 | 5.0E4 | 80 | 15mm | | |
| 実施例 5 (5) | 10 | 0 | 9.0E4 | 8.0E4 | 69 | 15mm | | |
| | 8 | 0 | 1.0E5 | 9.0E4 | 72 | 15mm | | |
| | 9 | 0 | 1.0E5 | 9.0E4 | 70 | 15mm | | |
| 比較例 1 | 比較例 1 | 500 | 200 | 10.0E5 | 12.0E5 | 20 | 15mm | |
| 比較例 2 | 比較例 2 | 100 | 50 | 8.0E5 | 9.0E5 | 30 | 15mm | |

[0036]

[Effect of the Invention] The silicon single crystal of this invention or the silicon single crystal by the manufacture approach of this invention reduction-izes the minute defect which becomes COP and a COP generating cause notably compared with the silicon single crystal raised by the approach learned conventionally, raises device properties, such as oxide film pressure-proofing, and fits the wafer for devices with which it is the outstanding crystal which makes a components separation percent defective reduction-ize, and high integration will progress further from now on. Moreover, the silicon single crystal manufacture approach of this invention reduction-izes the minute defect

which becomes COP and a COP generating cause notably, and raises device properties, such as oxide film pressure-proofing, and the outstanding crystal which makes a components separation percent defective reduction-ize can be offered.

[Translation done.]

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1. This document has been translated by computer. So the translation may not reflect the original precisely.
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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] (a) - (c) shows the relation of the volume density of the minute defect used as crystal cooling conditions, and the surface density of COP 0.11 micrometers or more, the generating situation of a R-OSF field and a COP generating cause, respectively.

[Drawing 2] (a) and (b) show the result corresponding to total amount change of the point defect which constitutes the whole minute defective product leading to [over the time amount at which a crystal stays change of the number of COP 0.11 micrometers or more to the time amount at which a crystal stays the temperature field 1300 degrees C or more at the time of carrying out crystal growth and a temperature field 1300 degrees C or more] COP generating, i.e., the minute defect concerned, from constant speed.

[Drawing 3] The CZ process silicon single crystal manufacturing installation which has the temperature controller 20 which anneals the crystal in the elevated-temperature field of 1300 degree C or more of **.

[Drawing 4] The CZ process silicon single crystal manufacturing installation which added temperature controller 30 another in order to anneal a 1100 degrees C - 1000 degrees C low-temperature field to ****3 .

[Drawing 5] The CZ process silicon single crystal manufacturing installation which added crystal cooling system 40 another in order to quench a 1100 degrees C - 1000 degrees C low-temperature field to ****3 .

[Drawing 6] The crystal cooling temperature pattern at the time of crystal pulling used in ***** and the example of a comparison is shown.

[Description of Notations]

- 1 -- CZ process silicon single crystal raising furnace
- 2 -- Wire loop wheel machine
- 3 -- Heat insulator
- 4 -- Heating heater
- 5 -- Rotation fixture
- 6 -- Crucible
- 6a -- Quartz crucible
- 6b -- Graphite crucible
- 7 -- Wire
- 8 -- Seed crystal
- 9 -- Chuck
- 10 -- Gas inlet
- 11 -- Gas exhaust
- 20 -- Temperature controller (crystal annealing equipment)
- 30 -- Temperature controller (crystal annealing equipment)
- 40 -- Temperature controller (crystal cooling system)

[Translation done.]

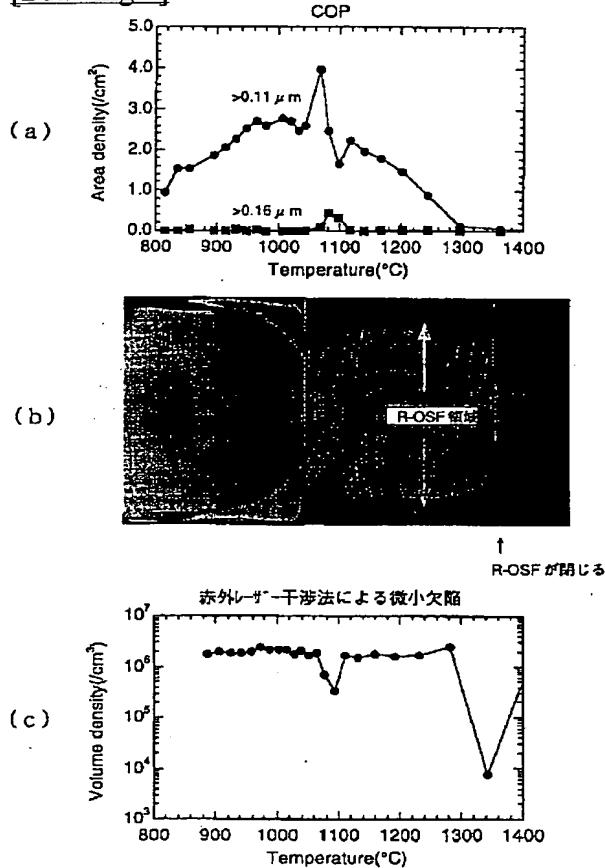
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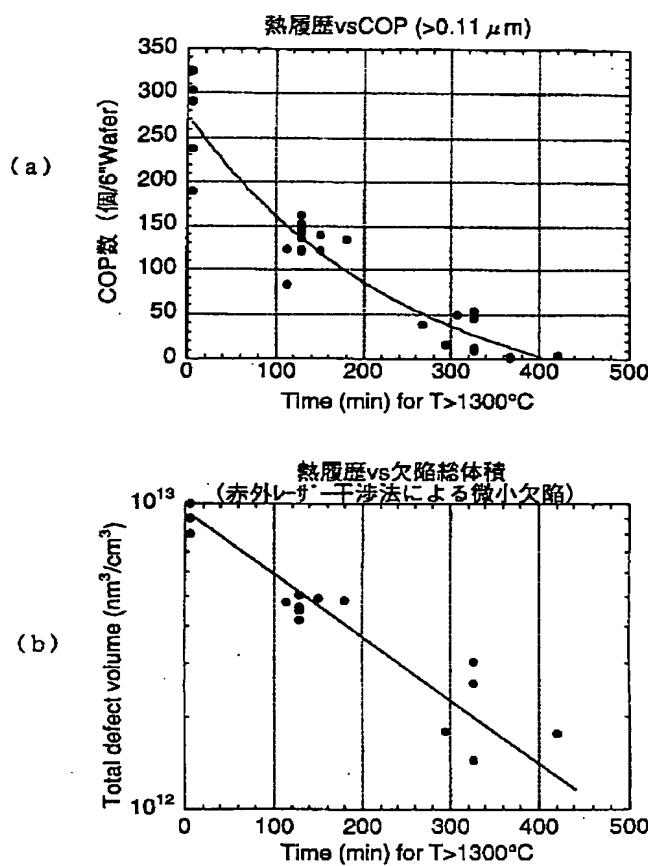
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DRAWINGS

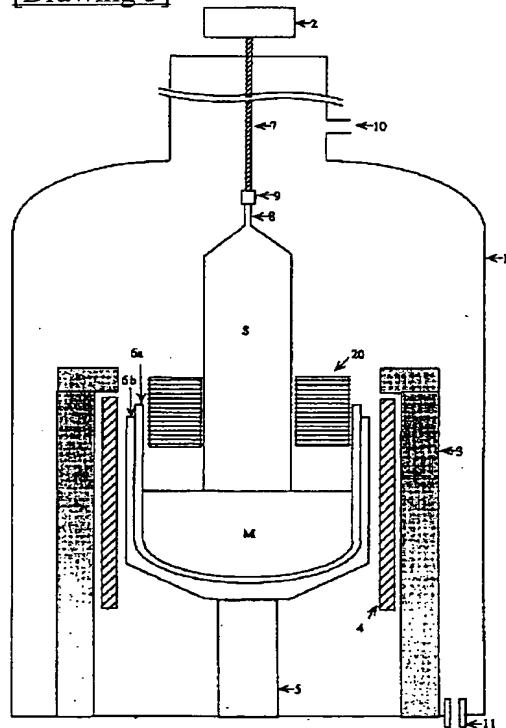
[Drawing 1]



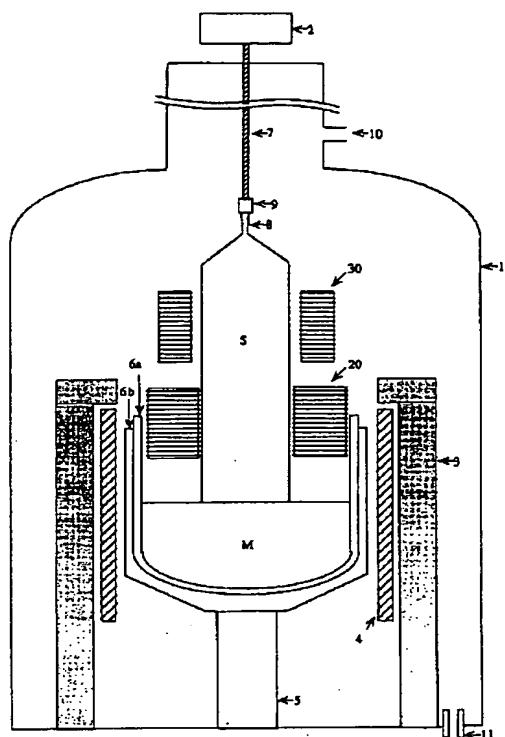
[Drawing 2]



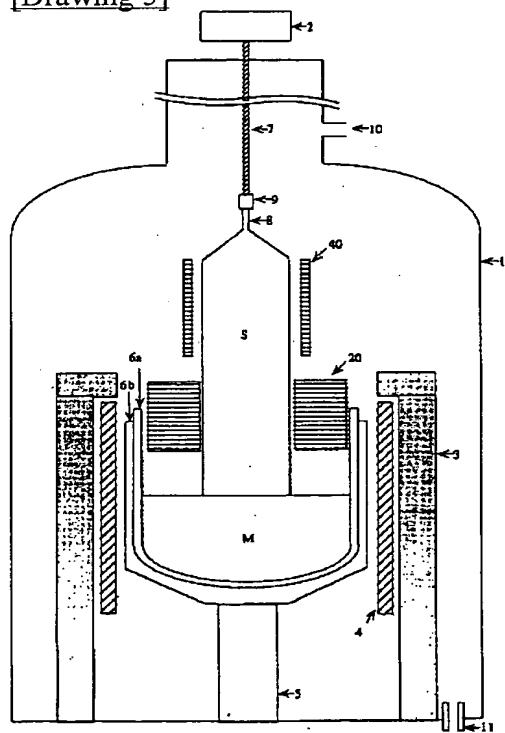
[Drawing 3]



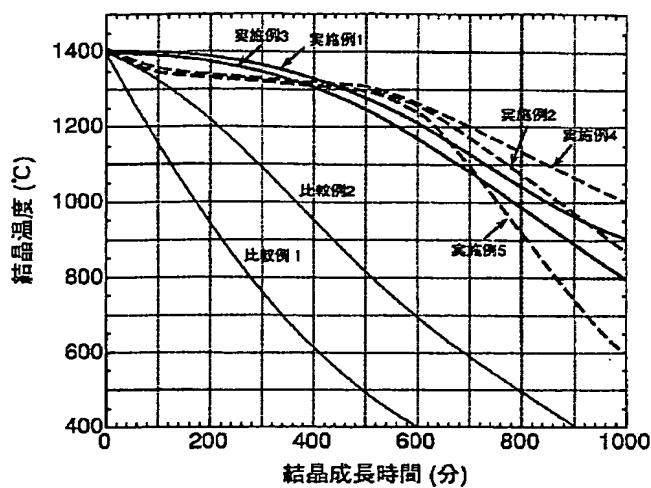
[Drawing 4]



[Drawing 5]



[Drawing 6]



[Translation done.]

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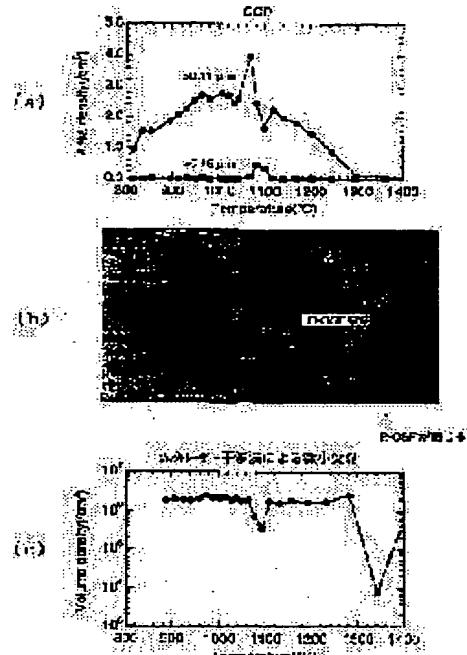
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(54) SILICON SINGLE CRYSTAL AND ITS PRODUCTION

(57)Abstract

PROBLEM TO BE SOLVED: To provide a method for pulling up a crystal by which crystal originated particles(COP) and microdefects to be a cause of producing the COP are reduced in the crystal produced by the Czochralski method.

SOLUTION: This method for producing a silicon single crystal comprises annealing a crystal in a temperature region of the melting point to $\geq 1,300^{\circ}$ C for ≥ 400 min and pulling up the crystal (method 1), regulating the time for passing a temperature region of $\geq 1,350^{\circ}$ C to <60 min in addition to the method 1 (method 2), carrying out the pulling up of the crystal under conditions so as to provide >0.13 ratio (v/G) of the pulling up speed (v: mm/min) to the crystal temperature gradient in the solidification interface (G: $^{\circ}$ C/mm) in addition to the method 1 (method 3), annealing the crystal at $<1.0^{\circ}$ C/min crystal cooling rate in a temperature region of 1,100 to 1,000 $^{\circ}$ C in addition to the method 1 or 2 (method 4) and quenching the crystal at $\geq 1.0^{\circ}$ C/min crystal cooling rate in the temperature region of 1,100 to 1,000 $^{\circ}$ C in addition to the method 1, 2 or 3 (method 5).



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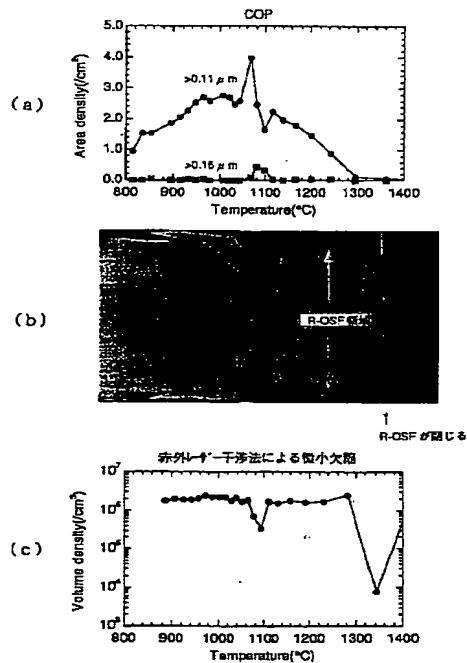
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(54) 【発明の名称】 シリコン単結晶およびその製造方法

(57) 【要約】 (修正有)

【課題】 チョクラルスキー法により製造される結晶において、COPおよびCOP発生原因となる微小欠陥を低減させる結晶引上げ方法を提供する。

【解決手段】 結晶を融点から1300°C以上の温度領域を400分以上徐冷して引き上げる(方法1)、方法1に加えて1350°C以上の温度領域を通過する時間を60分未満とする(方法2)、方法1に加えて引き上げ速度(v: mm/分)と凝固界面の結晶温度勾配(G: °C/mm)の比v/Gが0.13よりも大きくなる条件で結晶引上げを行う(方法3)、方法1あるいは方法2あるいは方法3に加えて1100°Cから1000°Cの温度領域を結晶冷却速度1.0°C/分未満で徐冷する(方法4)、方法1あるいは方法2あるいは方法3に加えて、1100°Cから1000°Cの温度領域を結晶冷却速度1.0°C/分以上で急冷する(方法5)より成る。



【特許請求の範囲】

【請求項1】 チョクラルスキー法によるシリコン単結晶の製造において、融点から1300°Cの結晶温度領域内を通過する時間が400分以上である結晶引き上げ成長をすることを特徴とするシリコン単結晶の製造方法。

【請求項2】 チョクラルスキー法によるシリコン単結晶の製造において、融点から1350°Cの結晶温度領域の通過時間が60分未満、1350°C～1300°Cの結晶温度領域内の通過時間が400分以上である結晶引き上げ成長をすることを特徴とするシリコン単結晶の製造方法。

【請求項3】 チョクラルスキー法によるシリコン単結晶の製造において、引上げ速度v (mm/分)、凝固界面での結晶温度勾配G (°C/mm) が、 $v/G > 0.13$ となる条件で引上げ、かつ1300°C以上の結晶温度領域内の通過時間が400分以上である結晶引き上げ成長をすることを特徴とするシリコン単結晶の製造方法。

【請求項4】 1100°Cから1000°Cの温度範囲の冷却速度が1.0°C/分未満であることを特徴とする請求項(1)および(2)に記載の結晶製造方法。

【請求項5】 1100°Cから1000°Cの温度範囲の冷却速度が1.0°C/分以上であることを特徴とする請求項(1)および(2)に記載の結晶製造方法。

【請求項6】 請求項(1)ないし(2)ないし(3)ないし(5)の方法で製造されたシリコン単結晶であって、0.13μm以上の大きなCOPが存在しないシリコン単結晶。

【請求項7】 請求項(1)ないし(2)ないし(3)ないし(4)ないし(5)の方法で製造されたシリコン単結晶から切り出され鏡面加工されたウエハーにおいて、0.11μm以上のCOPの表面密度が0.1個/cm²以下であるシリコン単結晶。

【請求項8】 請求項(1)ないし(2)ないし(3)ないし(4)ないし(5)の方法で製造されたシリコン単結晶において、COPを形成する微小結晶欠陥の体積密度が 5×10^5 個/cm³以下であるシリコン単結晶。

【請求項9】 請求項(1)ないし(2)ないし(3)ないし(4)ないし(5)の方法で製造されたシリコン単結晶であって、COPを形成する微小結晶欠陥体積が、母相の単位体積(1cm³)に対し 10^{12} nm³以下であるシリコン単結晶。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、結晶欠陥を大幅に低減させたチョクラルスキー法(以下、CZ法)により製造されたシリコン単結晶およびその製造法に関する。

【0002】

【従来の技術】CZシリコン単結晶は結晶強度が高いなどのすぐれた特徴を有しているため、従来よりLSI用の材料として広く用いられている。近年のMOSデバイ

ス集積度の増大とともに、ゲート酸化膜の信頼性向上やPN接合リーキ低減等のデバイス特性に優れ、また素子間分離における不良が生じにくいCZシリコン単結晶の製造技術開発が望まれていた。特に、COPは鏡面研磨後のシリコンウエハー表面に存在する微小ビットで、このビット自身およびビットを発生させる原因となる微小欠陥が先に述べたデバイス特性や素子間分離の不良原因となることが最近明らかにされ、その低減が切望されていた。

【0003】COP (Crystal Originated Particle) とは、シリコンウエハー加工工程において研磨後、ウエハー表面をアンモニア水と過酸化水素水の混合液(アンモニア:過酸化水素:水=1:1:5)で洗浄(SC-1洗浄と呼ばれる)した後に、ウエハー表面に微小欠陥に起因したビットが形成し、レーザーパーティクルカウンターで測定すると真性のパーティクルとともにパーティクルとして検出される。このようなビットを真性パーティクルと区別するためにCOPと呼ばれる。この低減方法については、従来、CZシリコン単結晶製造における酸化膜耐圧の向上と関係してその低減技術が知られていた。特開平6-27918号公報においては、1200°C～1420°Cの温度領域に1時間以上保持して結晶引上げする方法が開示され、特開平8-2993号公報では、1200°Cまでの高温域を通過する時間が200分以上となるようにし、1200°C～1000°Cの低温域を通過する時間が150分以下とする方法、特開平8-157293号公報では、1200°Cまでの高温域を通過する時間を200分未満とし1200°C～1000°Cの低温域を通過する時間を130分以下とする方法が開示されている。

【0004】これらの従来技術においては、本発明者らは、引上げ中の結晶の冷却条件を極端に変更した種々の結晶を育成し、また、引上炉内で結晶を保持する実験と共に研磨・洗浄直後のウエハーのCOP観察のみならず、洗浄と観察を繰り返し行い真性パーティクルを除去しCOP発生原因の欠陥のみの体積密度を評価する方法、および赤外レーザ干渉法によりCOP発生原因の微小欠陥の体積密度のみならずサイズも同時測定しCOP発生原因となっている微小欠陥を構成する点欠陥の総量を評価する観察実験にもとづいて、COPおよびCOP発生原因となっている微小欠陥の低減には1200°C～1300°Cの温度領域の保持では効果がなく、さらに、その温度領域における結晶通過時間も60～200分程度ではCOPおよびCOP発生原因の微小欠陥の顕著な低減は見られないことを見いたした。

【0005】したがって、引上げ中の結晶において顕著にCOPおよびCOP発生原因となる微小欠陥を低減させる結晶引上げ方法、さらに、現在市場において流通している大部分を占める、R-OSF (Ringlike distributed Oxidation-induced-Stacking-Faults: リング状

分布積層欠陥、参考文献：雑誌「応用物理」57巻、1541頁、1988年）領域が面内に存在せずエッジ部で消失している結晶あるいはR-OSF領域がウエハー中心で消失していない結晶に関して、COPおよびCOP発生原因となる微小欠陥を極端に低減する最適方法は存在しなかった。また、R-OSF領域がウエハー面内に存在せず、またウエハー中心でも消失していない結晶において、COPおよびCOP発生原因となる微小欠陥を極端に低減させた結晶も存在しなかった。

【0006】

【発明が解決しようとする課題】本発明は、チョクラルスキー法（以下、CZ法）により製造される結晶において、顕著にCOPおよびCOP発生原因となる微小欠陥を低減させる結晶引上げ方法、さらに、現在市場において流通している大部分を占める、R-OSF領域が面内に存在せずエッジ部で消失している結晶あるいはR-OSF領域がウエハー中心で消失していない結晶に関して、COPおよびCOP発生原因となる微小欠陥を極端に低減させたシリコン単結晶およびその製造法に関する。

【0007】

【課題を解決するための手段】上記目的を達成するためには、引上げ中の結晶に関して、結晶温度が1300°C以上の領域で、400分以上の保持される条件で結晶引上げ成長を行う（方法1）。さらに、R-OSF領域を結晶エッジ部外に除去しCOPおよびCOP発生原因の微小欠陥を低減するため融点から1350°Cの結晶温度領域の通過時間を60分未満とし、1350°C～1300°Cの結晶温度領域を400分以上保持する結晶引上げを行う（方法2）。あるいはR-OSF領域を結晶エッジ部外に除去しCOPおよびCOP発生原因の微小欠陥を低減するため、引上げ速度をv（mm/分）とし凝固界面での結晶温度勾配G（°C/mm）とするとき、v/G > 0.13となる条件で引上げ、かつ1300°C以上の結晶温度域内を通過する時間が400分以上となるよう結晶引き上げ成長する（方法3）。さらに、（方法1）あるいは（方法2）あるいは（方法3）に加えて、COPおよびCOP発生原因の微小欠陥密度をさらに小さくするために、1100°Cから1000°Cの温度範囲の冷却速度を1.0°C/分未満とし徐冷する（方法4）。また、（方法1）あるいは（方法2）あるいは（方法3）に加えて、COPおよびCOP発生原因の微小欠陥サイズをさらに小さくするために1100°Cから1000°Cの温度範囲の冷却速度を1.0°C/分以上とし急冷する（方法5）。

【0008】本発明方法1ないし2ないし3ないし5の方法で製造することによって0.13μm以上の大きなCOPが存在しないシリコン単結晶が得られる。また、本発明方法1ないし2ないし3ないし4ないし5の方法によって、シリコン単結晶から切断および鏡面加工した

ウエハー表面において0.11μm以上のCOPが0.1個/cm²以下であるシリコン単結晶を製造できる。本発明の1～5の製造方法によると、シリコン単結晶中に存在するCOPを形成する微小結晶欠陥の体積密度は5×10⁵/cm³以下であり、また、当該微小欠陥を構成する点欠陥濃度が、母相の単位体積（1cm³）に対し10¹²nm³以下である通常従来結晶に比べて1桁以上微小欠陥密度および点欠陥濃度を低減したシリコン単結晶が得られ、絶縁酸化膜の耐電圧特性やP/Nリード特性などのデバイス特性も優れている。

【0009】

【発明の実施の形態】本発明者らは、引上げ中の結晶の冷却条件を極端に変更した種々の結晶を育成し、また、引上炉内で結晶を保持する実験と単に研磨・洗浄直後のウエハーのCOP観察のみならず、洗浄と観察を繰り返し行い真性パーティクルを除去しCOP発生原因の欠陥のみの体積密度を評価する方法、および赤外レーザ干渉法によりCOP発生原因の微小欠陥の体積密度のみならずサイズも同時測定しCOP発生原因となっている微小欠陥を構成する点欠陥の総量を評価する観察実験にもとづいて、COPおよびCOP発生原因となる微小欠陥の形成と結晶冷却条件の間に次のような関係があることを発見した。すなわち、引上げ中の結晶を引上げ途中で引上げ速度を極端にほぼ停止状態まで減速し引上炉内で結晶を保持しCOPおよびCOP発生原因となる微小欠陥の発生挙動を調べたところ、図1に示すような結果となった。図1(a)には0.11μm以上のCOPの面密度、図1(b)にはR-OSF領域を示すためのX線トボグラフ写真、そして図1(c)には赤外レーザー干渉法により測定したCOP発生原因となる微小欠陥の体積密度をそれぞれ引上げ中の結晶の保持温度の関数として示している。図1(a)および(c)から明らかなように、COPおよびCOP発生原因となる微小欠陥が面密度でゼロレベル、体積密度で10⁴/cm³以下に減少する温度領域は1300°C以上である。また、図1(b)のX線トボグラフ写真において、融点から1350°Cの温度領域を保持された部位は、R-OSF領域が結晶中心部で閉じ消失しているのが明らかである。したがって、COPあるいはCOPを発生させる原因となる微小欠陥を低減させるためには1300°C以上の徐冷が必要であるが、1350°C以上を徐冷しすぎるとR-OSF領域が結晶中心側に存在するようになる。また、図1(c)において1100°C～1000°Cの温度範囲において体積密度が減少するのが認められ、図1(a)では、同温度範囲において0.11μm以上のCOP面密度の一時的な減少と一時的な増加が認められる。0.11μm以上のCOP面密度が一時的に減少する位置は0.16μm以上の大きなCOPが増加する位置と一致していることから、この温度領域ではCOP発生原因となる微小欠陥が成長することがわかる。特に、大きな微

小欠陥はより高温側で成長し小さな微小欠陥は低温側で成長する。この結果、0.11 μm以上のCOPの一時的な増加は、小さなCOP発生原因微小欠陥が成長した領域であると考えられる。この結果から、この温度領域の冷速を高めることによりCOP発生原因となる微小欠陥の成長を抑制することが可能で、また、この温度領域を徐冷することで、微小欠陥密度をさらに低減することが可能である。

【0010】上記は、引上炉内における引上げ結晶の保持による微小欠陥観察結果であったが、実際に引上炉内で引上げ中の結晶を減速し保持することなく、一定速度で結晶成長した場合の高温域の徐冷効果を調べたところ、図2に示す結果を得た。すなわち、図2(a)は1300°C以上の温度領域を通過する結晶の滞在時間に対する0.11 μm以上のCOPの個数の変化を示し、図2(b)は1300°C以上の温度領域を通過する結晶の滞在時間に対するCOP発生原因となる微小欠陥の総体積、すなわち当該微小欠陥を構成する点欠陥の総量変化に対応する結果を示す。この結晶においてはR-OSF領域は結晶エッジ部に位置し中心領域に存在しない。図2から明らかなように、1300°C以上の温度領域を400分以上通過することによってCOPはほぼゼロレベルで、COPを発生させる微小欠陥構成要素である点欠陥の総量がほぼ1桁減少することがわかる。

【0011】以上の結果から、COPおよびCOP発生原因となる微小欠陥の形成を抑制するためには高温徐冷が必要であるが、効果的な温度範囲は1300°C以上であり必要とする徐冷時間は400分以上である。

【0012】さらに、現在市場において流通している大部分を占める、R-OSF領域が面内に存在せずエッジ部で消失している結晶あるいはR-OSF領域がウェハーセンターで消失していない結晶の製造条件は以下の二つの条件であることがわかった。すなわち、徐冷温度を1350°C以上にすると、徐冷時間にともなってR-OSF領域が結晶エッジ部から中心側に入り込むようになる。本発明においては、R-OSF領域を結晶中心部で消失させない条件として1350°C以上の徐冷時間は60分未満が好ましいことが明らかとなった。R-OSF領域が結晶中心部において消失しない別の条件として、引上げ速度(v: mm/分)と凝固界面の結晶温度勾配

(G: °C/mm)の比v/Gが0.13よりも大きくなる条件で結晶引上げを行えばよいことがわかった。この引上げ速度(v: mm/分)と凝固界面の結晶温度勾配

(G: °C/mm)の比v/Gによって、R-OSF領域の発生位置が変化することは特開平7-257991号公報に記載されているが、本発明においては、R-OSF領域が結晶中心部において消失しない結晶引上げ条件はv/Gが0.13よりも大きくなる条件であることがわかった。

【0013】COP欠陥制御のための低温側の結晶冷却

条件に関しては、その効果は特開平8-2993号公報において、COP発生原因となる微小欠陥の成長に関係し冷速が大きい方が当該微小欠陥の成長が抑制され、冷速を小さくすることで成長が進むが密度を低減することができる事が開示されている。しかしながら、本発明においては、1300°C以上の高温徐冷を十分行っておけば1100°C~1000°Cの低温度領域においての急冷速度が1.0°C/分程度でもCOPおよびCOP発生原因となる微小欠陥は十分に低減できることが初めて見いだされた。さらに、1300°C以上の高温徐冷を十分に行っておけば、1100°C~1000°Cの低温度領域において1.0°C/分以下の冷速で徐冷を行うことにより大きなCOP発生原因となる微小欠陥は成長せず、むしろ一層の密度低下をもたらすことも初めて見いだされた。また、本発明の1300°C以上の十分な高温徐冷と1100°C~1000°Cの低温度領域の急冷(冷却速度が1.0°C/分以上)を組み合わせることによってCOPサイズの低減によるCOP発生の抑制効果が顕著となることは言うまでもない。1100°C~1000°Cの温度領域の効果は、冷却速度が1.0°C/分未満の徐冷の場合はCOPおよびCOP発生原因となる微小欠陥の構成要素である点欠陥の凝集を促進することによりCOPおよびCOP発生原因の微小欠陥密度を低減させる効果があり、冷却速度が1.0°C/分以上の急冷の場合は、逆にCOPおよびCOP発生原因となる微小欠陥の構成要素である点欠陥の凝集を抑制することによりCOPおよびCOP発生原因の微小欠陥のサイズを低減させる効果がある。

【0014】本発明方法1では、結晶引き上げ炉内に温度制御機能を設置し1300°C以上の温度領域において、引上げられる結晶を400分以上徐冷することでCOPおよびCOP発生原因となる微小欠陥を構成する主たる点欠陥である原子空孔を結晶外方へ拡散させ減少させることにより、当該微小欠陥の形成を低減する。

【0015】本発明方法2では、方法1の作用に加えて、R-OSF領域が結晶中心側に入り込まないようにするために1350°C以上の温度領域を通過する時間を60分未満とする。

【0016】本発明方法3では、方法1の作用に加えて、R-OSF領域が結晶中心側に入り込まないようにするために引上げ速度(v: mm/分)と凝固界面の結晶温度勾配(G: °C/mm)の比v/Gが0.13よりも大きくなる条件で結晶引上げを行う。

【0017】本発明方法4では、方法1あるいは方法2あるいは方法3の作用に加えて、さらにCOPおよびCOP発生原因となる微小欠陥密度を低減させるために1100°Cから1000°Cの温度領域を結晶冷却速度1.0°C/分未満で徐冷する。

【0018】本発明方法5では方法1あるいは方法2あるいは方法3の作用に加えて、さらにCOPおよびCOP

P発生原因となる微小欠陥サイズを低減させるために100°Cから1000°Cの温度領域を結晶冷却速度1.0°C/分以上で急冷する。

【0019】

【実施例】以下に本発明の実施例を挙げて説明するが、本発明がこれらの実施例の記載によって制限されるものでないことは言うまでもない。

【0020】実施例に先立ち、本発明により製造したシリコン単結晶のCOPおよびCOP発生原因となる微小欠陥、さらにR-OSF領域の有無の評価方法、またはデバイス特性評価方法を以下に説明する。

【0021】COPは、製造した結晶をウエハー加工し鏡面研磨後、アンモニア：過酸化水素：水=1:1:5のSC-1洗浄液で洗浄した後、レーザーパーティクルカウンターLS6000でパーティクル数を測定した。さらに、真性パーティクルを除去しCOP発生原因の欠陥のみの体積密度を評価するためにSC-1洗浄とパーティクル測定を10回繰り返し、その増分から体積密度を求めた。COP発生原因となる微小欠陥は、赤外レーザ干渉法による測定装置(OPP: Oxygen Precipitate Profiler)により密度、サイズを計測した。R-OSF領域の形成の有無については、OSFを発生させるために1100°Cで酸化雰囲気中で90分熱処理後、X線トポグラフにより観察した。

【0022】酸化膜耐圧は鏡面加工を施したシリコンウェーハ試料上にMOSダイオードを形成し、1000°Cの乾燥酸素雰囲気中で形成された25.0nmの二酸化珪素膜であるゲート酸化膜(絶縁酸化膜)の電気特性を調べることによって行った。酸化膜を通して流れる電流密度が1μA/cm²の時のゲート酸化膜に印加される平均電界を測定した。特に、その平均電界が8.0MV/cm以上である場合は、真性絶縁破壊領域(Cモード領域)と呼ばれる耐圧劣化を起こす結晶欠陥が存在しない領域である。したがって、耐圧特性評価において、平均電界が8.0MV/cm以上で破壊する(Cモード領域にある)MOSダイオードの個数の総数に対する割合が多いシリコンウェーハが耐圧特性の優れた結晶である。

【0023】以下に、実施例および比較例を述べるが、引上げ育成した結晶の仕様は、伝導型:p型(ボロンドープ)、結晶径:6インチ用(直径160mm)、抵抗率:10Ω·cm、酸素濃度:8.5~9.5×10¹⁷atoms/cm³(日本電子工業振興協会による酸素濃度換算係数を用いて算出)、炭素濃度:<1.0×10¹⁶atoms/cm³(日本電子工業振興協会による炭素濃度換算係数を用いて算出)である。

【0024】表1に、本発明の実施例1から実施例5、および比較例1、比較例2におけるウェーハのレーザーパーティクルカウンターLS6000で測定したCOP、赤外レーザ干渉法(OPP)にて計測したCOP発生原因となる微小欠陥の体積密度、および酸化膜耐圧、

さらにR-OSFの存在位置それぞれの測定結果のまとめを示す。

【0025】実施例1

本発明に用いられるシリコン単結晶製造装置は、通常チョクラルスキー法によるシリコン単結晶製造に用いられるものであれば特に限定されるものではなく、本実施例では図3に示すような製造装置を用いた。このチョクラルスキー法シリコン単結晶製造装置の特徴は、結晶冷却温度および速度パターンが図6中(実施例1)のような引き上げ条件、結晶引き上げ炉内で1300°C以上の温度域を400分以上経過して結晶が通過するような徐冷領域を形成するために温度制御装置を設置した。温度制御装置としては、引き上げ育成されるシリコン単結晶インゴットを取り囲むように設置された黒鉛などの断熱保温材や加熱ヒータなどが有効である。

【0026】この装置を使用して、以下の条件でシリコン単結晶を育成した。この単結晶インゴットから切りだしたウェーハのCOPおよび赤外レーザ干渉法(OPP)にて計測したCOP発生原因となる微小欠陥の体積密度、酸化膜耐圧、R-OSFの存在位置を表1に示した。これらのシリコンウェーハにおいては、0.13μm以上のCOPはゼロレベルで、0.11μm以上の小さなCOPを含めても約10個程度(0.05個/cm²)で従来技術に比べて1桁程度低減されている。また、COPおよびCOP発生原因となる微小欠陥の体積密度は1.0×10⁵/cm³以下でやはり1桁低減されている。結果的に耐圧特性も非常に良好である。本結晶においては1350°C以上の徐冷の効果のためにR-OSF領域が若干エッジから内側に入っている。

【0027】実施例2

実施例1の装置を用いて、以下の条件でシリコン単結晶を育成した。結晶冷却温度および速度パターンが図6中(実施例2)のような引き上げ条件、すなわち1350°C以上の温度領域の通過時間は60分未満であるが、1350°C~1300°Cの結晶温度領域を400分程度経過しゆっくり冷却されるような条件でシリコン単結晶を育成した。この単結晶インゴットから切りだしたウェーハのCOPおよび赤外レーザ干渉法(OPP)にて計測したCOP発生原因となる微小欠陥の体積密度、酸化膜耐圧、R-OSFの存在位置を表1に示した。これらのシリコンウェーハにおいても実施例1と同様、0.13μm以上のCOPはほぼゼロレベルで、0.11μm以上の小さなCOPを含めても20個(0.1個/cm²)以下で従来技術に比べて1桁程度低減されている。また、COPおよびCOP発生原因となる微小欠陥の体積密度は1.0×10⁵/cm³レベルで顕著に低減されている。結果的に耐圧特性も非常に良好である。本結晶においては1350°C以上の保持時間を60分未満としたためにR-OSF領域は結晶インゴットエッジないしはエッジより外方に消滅している。

【0028】実施例3

実施例1の装置を用いて、以下の条件でシリコン単結晶を育成した。引上げ速度 v (mm/分) と凝固界面での結晶側の温度勾配 G (°C/mm) の比 v/G が 0.13 より大きくなるようにした。すなわち、本実施例においては凝固界面での結晶温度勾配は約 1.8 °C/mm で、引上げ速度 v を 0.6 mm/分に設定し（この場合 $v/G = 0.33$ ）結晶引上げを実施した。さらに、1300 °C 以上の結晶温度領域を 400 分程度経過しゆっくり冷却されるような条件でシリコン単結晶を育成した。この場合の結晶冷却温度および速度パターンは図 6 中（実施例 3）で、実施例 1 に比べて引上げ速度が高めのため高温での徐冷時間が短くなっている。しかしながら、凝固界面付近の断熱や加温条件を変更することにより温度勾配を本実施例よりさらに小さくしたり、高温領域を延長する事が可能であり徐冷時間は長くすることができる。本実施例にて引上げ育成した単結晶インゴットから切りだしたシリコンウェーハの COP 数、繰り返し洗浄により求めた COP 体積密度、赤外レーザー干渉法 (OPP) により求めた COP 発生原因となる微小欠陥密度、酸化膜耐圧の測定結果、表 1 に示す。これらのシリコンウェーハにおいても実施例 1、2 と同様、0.13 μm 以上の COP はほぼゼロレベルで、0.11 μm 以上のような小さな COP を含めても 20 個 ($0.1\text{個}/\text{cm}^2$) 以下で従来技術に比べて 1 衍程度低減されている。また、COP および COP 発生原因となる微小欠陥の体積密度は $1.0 \times 10^5 / \text{cm}^3$ レベルで顕著に低減されている。結果的に耐圧特性も非常に良好である。本結晶においては R-O SF 領域は結晶インゴットエッジより外方に消滅している。

【0029】実施例4

本実施例では図 4 に示すような製造装置を用いた。すなわち、実施例 1 および図 3 で示した製造装置に加えて、さらに 1100 °C から 1000 °C の低温温度領域も 1.0 °C/分以下の冷却速度で徐冷するために図 3 に示した温度制御装置の上方に、さらに保温断熱材や加温ヒータで構成される保温あるいは加温装置を設置した。なお、図 4 のように新たに保温あるいは加温装置を設置しなくとも、図 3 の温度制御装置を上方、すなわち結晶温度が低い側に延長するのでもよい。このような製造装置を用い、1300 °C 以上の結晶温度領域を 400 分以上保持した後、さらに 1100 °C から 1000 °C の温度領域を 1.0 °C/分以下の冷却速度で徐冷し結晶引上げを行った。このシリコン単結晶インゴットから切りだしたウェーハの COP 数、繰り返し洗浄により求めた COP 体積密度、赤外レーザー干渉法 (OPP) により求めた COP 発生原因となる微小欠陥密度、酸化膜耐圧の測定結果、表 1 に示す。これらのシリコンウェーハにおいては、0.13 μm 以上の COP も 0.11 μm 以上のような小さな COP も 10 個 ($0.05\text{個}/\text{cm}^2$) 以下で低減

効果が著しい。しかしながら、1100 °C から 1000 °C の徐冷効果により 0.13 μm 以上の大きな COP の若干の増加が見られるのが特徴である。また、COP および COP 発生原因となる微小欠陥の体積密度も $5.0 \sim 8.0 \times 10^4 / \text{cm}^3$ レベルで最も低くなっている。その結果、耐圧特性も非常に良好である。本結晶においては R-O SF 領域は結晶インゴットエッジあるいはエッジ外方に消滅している。

【0030】実施例5

10 本実施例では図 5 に示すような製造装置を用いた。すなわち、実施例 1 および図 3 で示した製造装置に加えて、さらに 1100 °C から 1000 °C の低温温度領域を 1.0 °C/分以上の冷却速度で冷却するため図 3 に示した温度制御装置の上方に、冷却速度を高めるために円筒型の冷却装置を設置した。冷却装置としては、熱伝導性の良く輻射率の大きい黒鉛板や金属板が有効で、当該冷却装置によって結晶からの輻射熱を抜熱し、さらに当該冷却装置と引上げ途中の結晶の間を流れる雰囲気ガス（本実施例ではアルゴンガス）による対流伝熱による抜熱効果で冷却される。当該冷却装置として用いられる黒鉛板や金属板をガスや液体を用いて強制冷却してもよい。このような製造装置を用い、1300 °C 以上の結晶温度領域を 400 分以上保持した後、さらに 1100 °C から 1000 °C の温度領域を 1.0 °C/分以上の冷却速度で冷却し結晶引上げを行った。このシリコン単結晶インゴットから切りだしたウェーハの COP 数、繰り返し洗浄により求めた COP 体積密度、赤外レーザー干渉法 (OPP) により求めた COP 発生原因となる微小欠陥密度、酸化膜耐圧の測定結果、表 1 に示す。これらのシリコンウェーハにおいては、0.13 μm 以上の COP はゼロで、0.11 μm 以上のような小さな COP も 10 個 ($0.05\text{個}/\text{cm}^2$) 以下で低減効果が著しい。また、COP および COP 発生原因となる微小欠陥の体積密度も $1.0 \times 10^5 / \text{cm}^3$ レベルで顕著に密度が低減されている。その結果、耐圧特性も非常に良好である。本結晶においては R-O SF 領域は結晶インゴットエッジあるいはエッジ外方に消滅している。

【0031】比較例1

40 本比較例は、従来技術の一例である。図 3 に示した実施例 1 で用いた製造装置と同様な結晶成長装置を用いた。しかしながら、結晶冷却温度および速度パターンは図 6 中（比較例 1）のような引き上げ条件、すなわち、結晶引き上げ炉内で 1300 °C 以上の温度領域に保持される時間は 40 分程度で、1200 °C 以上に保持される時間も 80 分程度であるような高温冷却条件を実現するよう温度制御装置を設置し、さらに 1200 °C ~ 1000 °C の温度領域通過時間が 80 分程度であるようなシリコン単結晶引上げ装置である。温度制御装置としては、引き上げ育成されるシリコン単結晶インゴットを取り囲むように設置された黒鉛などの断熱保温材や加熱ヒータなど

が有効である。ただし、実施例1と比較して高温領域の徐冷結晶長さあるいは時間が短いことが特徴である。本比較例の結晶冷却パターンは、高温領域の徐冷時間が100分程度以下で比較的短いことから冷却条件を制御する温度制御装置を必ずしも用いる必要はなく、結晶成長速度すなわち引上げ速度を低速にすることによっても実現可能である。

【0032】この引上げ装置を使用して、結晶成長したシリコン単結晶インゴットから切りだしたウェーハのCOPおよび赤外レーザ干渉法(OPP)にて計測したCOP発生原因となる微小欠陥の体積密度、酸化膜耐圧、R-O SFの存在位置を表1に示した。これらのシリコンウェーハにおいては、0.13μm以上のCOPは200個(1.0個/cm²)で、0.11μm以上の小さなCOPを含めると500個(2.5個/cm²)程度である。また、COPおよびCOP発生原因となる微小欠陥の体積密度は1.0×10⁶/cm³程度ある。耐圧特性は、その特性の指標である8.0MV/cm以上の印加電界で破壊する面積比率は20%程度である。これら本比較例の結果は、本発明の実施例に比べてCOPの個数および密度、あるいはCOP発生原因となっている微小欠陥の密度いずれも非常に悪い。また、酸化膜耐圧も良好ではない。

【0033】比較例2

本比較例では、比較例1で用いた装置と同様な装置を用いて結晶育成した。ただし、本比較例の結晶冷却温度お

より速度パターンは図6中(比較例2)のような引き上げ条件、すなわち1300°C以上の温度領域を通過する時間が120分程度で、1200°C以上の温度領域に保持される時間が220分程度であり、さらに1200°C～1000°Cの温度領域通過時間が100分程度であることが特徴である。

【0034】この引上げ装置を使用して、結晶成長したシリコン単結晶インゴットから切りだしたウェーハのCOPおよび赤外レーザ干渉法(OPP)にて計測したCOP発生原因となる微小欠陥の体積密度、酸化膜耐圧、R-O SFの存在位置を表1に示した。これらのシリコンウェーハにおいては、0.13μm以上のCOPは50個(0.25個/cm²)で、0.11μm以上のCOP数は100個(0.5個/cm²)程度であり、比較例1に比較してCOP数が低減している。また、COPおよびCOP発生原因となる微小欠陥の体積密度は8.0×10⁵/cm³～1.0×10⁶/cm³程度で、やはり比較例1に比べて若干減少が認められる。しかしながら、本発明で実現したようなCOP数がほぼゼロに近いレベル、あるいはCOP発生原因の微小欠陥の体積密度も1桁減少させ、耐圧特性も8.0MV/cm以上の破壊面積比率が60%以上である品質レベルからは、はるかに劣るものである。

【0035】

【表1】

| 冷却温度 n _{Ar} -7 | | >0.11 μmのCOP面数 G ^{0.11} μm | | >0.13 μmのCOP面数 G ^{0.13} μm | | 露凝結によるCOP面数 >0.11 μm / cm ² | | 赤外レーベ干渉法(OPI)による 欠陥密度 (>100nm / cm ³) | | 8.0NY/cm以上耐圧合格率 (%) | | R-OSF存在位置 | |
|-----------------------------------|-------|--|-----|--|--------|---|--------|--|----|-------------------------|--|-----------|--|
| 実施例 1 | (1) | 12 | 0 | 0 | 9.0E4 | 8.0E4 | 8.0E4 | 70 | 70 | x _{77'} ~ 15mm | | | |
| 実施例 2 | (2) | 11 | 0 | 8.0E4 | 8.0E4 | 8.0E4 | 8.0E4 | 68 | 68 | x _{77'} ~ 20mm | | | |
| 実施例 3 R-OSFが結晶内部に 存在しない引上条件 | (3) | 10 | 0 | 1.0E5 | 1.0E5 | 9.0E4 | 9.0E4 | 62 | 62 | x _{77'} ~ 15mm | | | |
| 実施例 4 | (4) | 16 | 2 | 1.2E5 | 1.1E5 | 1.1E5 | 1.1E5 | 78 | 78 | x _{77'} 外 | | | |
| 実施例 5 | (5) | 13 | 0 | 1.0E5 | 1.0E5 | 9.0E4 | 9.0E4 | 75 | 75 | x _{77'} 外 | | | |
| 実施例 6 | | 15 | 1 | 9.0E4 | 9.0E4 | 9.0E4 | 9.0E4 | 72 | 72 | x _{77'} 外 | | | |
| 実施例 7 | | 19 | 1 | 1.2E5 | 1.0E5 | 1.0E5 | 1.0E5 | 65 | 65 | x _{77'} 外 | | | |
| 比較例 1 | | 16 | 0 | 1.0E5 | 9.0E4 | 9.0E4 | 9.0E4 | 63 | 63 | x _{77'} 外 | | | |
| 比較例 2 | | 18 | 1 | 1.0E5 | 8.0E4 | 8.0E4 | 8.0E4 | 70 | 70 | x _{77'} 外 | | | |
| 比較例 3 | | 6 | 3 | 8.0E4 | 8.0E4 | 8.0E4 | 8.0E4 | 83 | 83 | x _{77'} 外 | | | |
| 比較例 4 | | 7 | 4 | 5.0E4 | 5.0E4 | 5.0E4 | 5.0E4 | 85 | 85 | x _{77'} 外 | | | |
| 比較例 5 | | 5 | 3 | 6.0E4 | 5.0E4 | 5.0E4 | 5.0E4 | 80 | 80 | x _{77'} 外 | | | |
| 比較例 6 | | 10 | 0 | 9.0E4 | 8.0E4 | 8.0E4 | 8.0E4 | 69 | 69 | x _{77'} 外 | | | |
| 比較例 7 | | 8 | 0 | 1.0E5 | 9.0E4 | 9.0E4 | 9.0E4 | 72 | 72 | x _{77'} 外 | | | |
| 比較例 8 | | 9 | 0 | 1.0E5 | 9.0E4 | 9.0E4 | 9.0E4 | 70 | 70 | x _{77'} 外 | | | |
| 比較例 9 | 比較例 1 | 500 | 200 | 10.0E5 | 12.0E5 | 12.0E5 | 12.0E5 | 20 | 20 | x _{77'} 外 | | | |
| 比較例 10 | 比較例 2 | 100 | 50 | 8.0E5 | 9.0E5 | 9.0E5 | 9.0E5 | 30 | 30 | x _{77'} 外 | | | |

【0036】

【発明の効果】本発明のシリコン単結晶あるいは本発明の製造方法によるシリコン単結晶は、従来知られていた方法によって育成したシリコン単結晶に比べて顕著にCOPおよびCOP発生原因となる微小欠陥を低減化し、酸化膜耐圧などのデバイス特性を向上させ、素子間分離不良率を低減化させる優れた結晶であり今後さらに高集積化が進むデバイス用ウェーハに適する。また、本発明

のシリコン単結晶製造方法は、顕著にCOPおよびCOP発生原因となる微小欠陥を低減化し、酸化膜耐圧などのデバイス特性を向上させ、素子間分離不良率を低減化させる優れた結晶を提供できるものである。

【図面の簡単な説明】

【図1】(a)～(c)は、それぞれ、結晶冷却条件と0.11 μm以上のCOPの面密度、R-OSF領域の発生状況、COP発生原因となる微小欠陥の体積密度の

関係を示す。

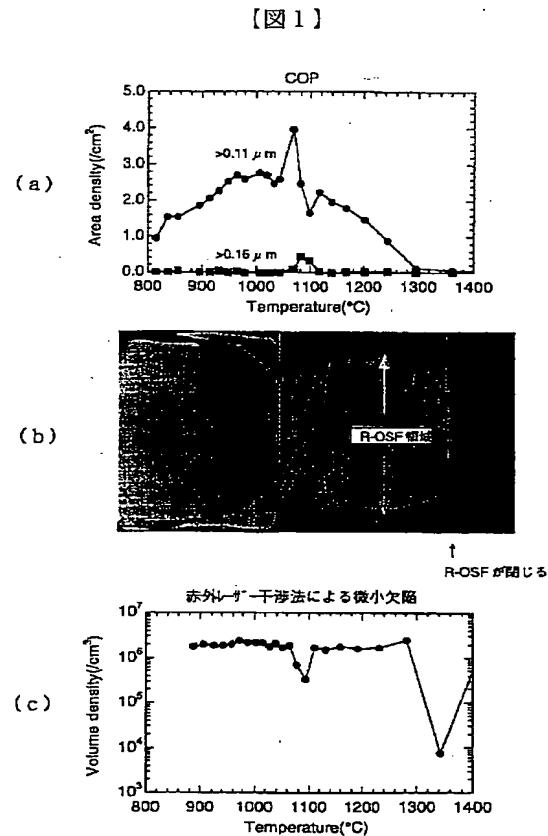
【図2】(a)、(b)は一定速度で結晶成長した場合の1300°C以上の温度領域を結晶が滞在する時間に対する0.11μm以上のCOPの個数の変化および1300°C以上の温度領域を結晶が滞在する時間に対するCOP発生原因となる微小欠陥の総体積、すなわち当該微小欠陥を構成する点欠陥の総量変化に対応する結果を示す。

【図3】は1300°C以上の高温領域での結晶を徐冷する温度制御装置20を有するCZ法シリコン単結晶製造装置。

【図4】は図3に1100°C~1000°Cの低温領域を徐冷するために別の温度制御装置30を付加したCZ法シリコン単結晶製造装置。

【図5】は図3に1100°C~1000°Cの低温領域を急冷するために別の結晶冷却装置40を付加したCZ法シリコン単結晶製造装置。

【図6】は実施例および比較例で用いた結晶引上時の結晶冷却温度パターンを示す。

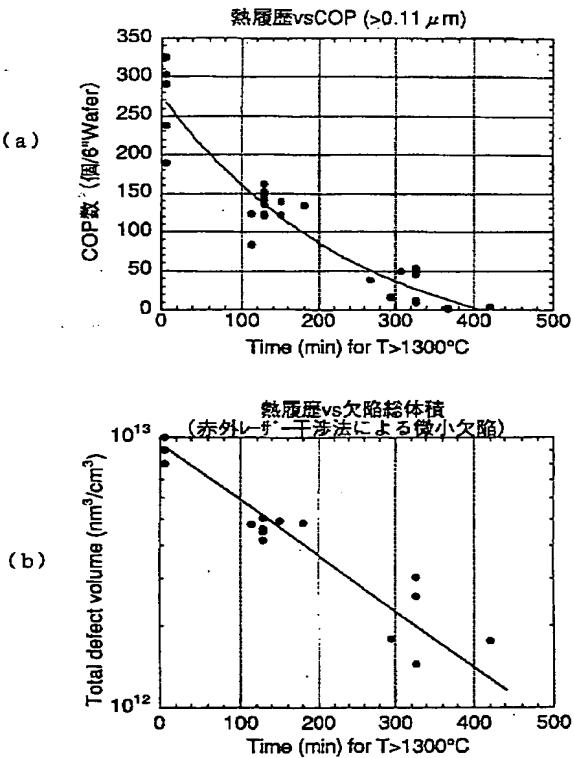


* 晶冷却温度パターンを示す。

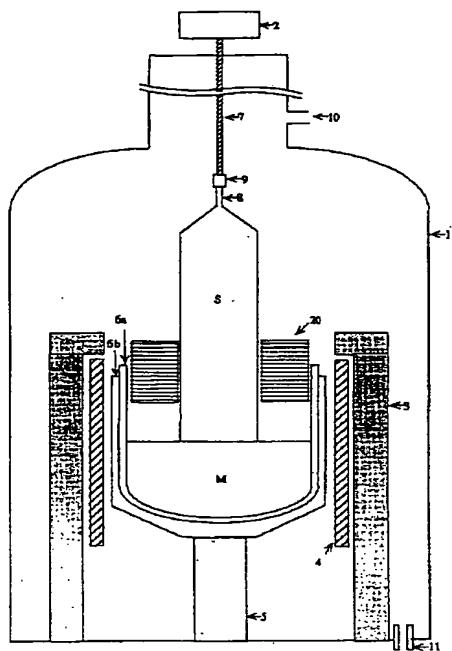
【符号の説明】

- 1 … CZ法シリコン単結晶引き上げ炉
- 2 … ワイヤ巻き上げ機
- 3 … 断熱材
- 4 … 加熱ヒータ
- 5 … 回転治具
- 6 … ルツボ
- 6 a … 石英ルツボ
- 6 b … 黒鉛ルツボ
- 7 … ワイヤ
- 8 … 種結晶
- 9 … チャック
- 10 … ガス導入口
- 11 … ガス排出口
- 20 … 温度制御装置（結晶徐冷装置）
- 30 … 温度制御装置（結晶徐冷装置）
- 40 … 温度制御装置（結晶冷却装置）

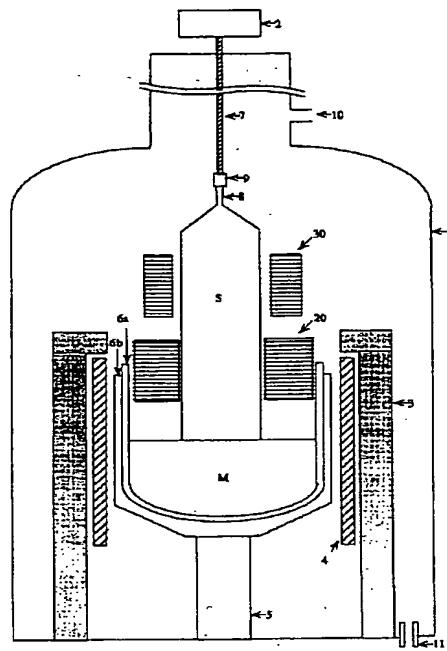
【図2】



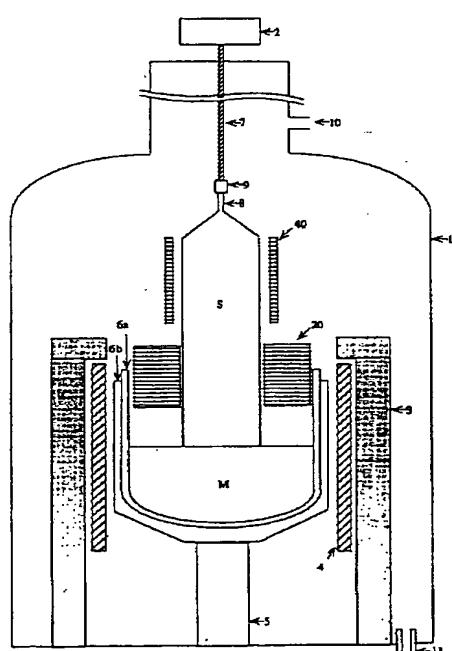
【図3】



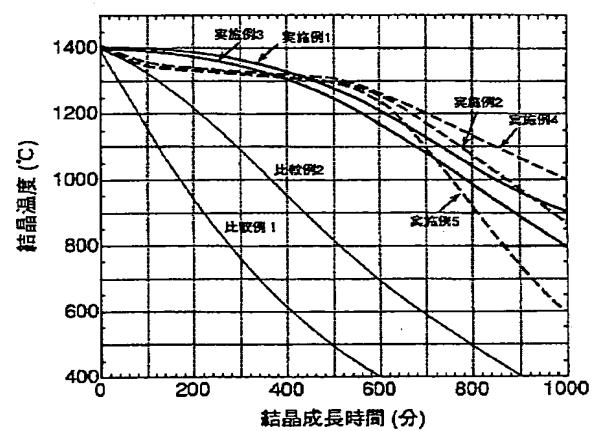
【図4】



【図5】



【図6】



フロントページの続き

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